भारतीय मानक
Indian Standard

IS 16955 : 2019

स्पिरोमेसिफेन, निस्पंदित सांद्र (एससी) — विशिष्टि

Spiromesifen, Suspension Concentrate (SC) — Specification

ICS 65.100.30

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Pesticides Sectional Committee, FAD 01

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Spiromesifen, Suspension Concentrate (SC) is generally used as an insecticide.

Spiromesifen, SC is generally manufactured to contain 229 g/kg or 22.9 percent (*m/m*) of Spiromesifen.

In the formulation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPIROMESIFEN, SUSPENSION CONCENTRATE (SC) — SPECIFICATION

1 SCOPE

This standard specifies the requirements and methods of sampling and test for Spiromesifen, Suspension Concentrate (SC).

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
6940 :1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 2) : 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (*second revision*)
9754 : 1981	Specification for high density polyethylene containers for packing of liquid pesticides (up to 1 Litre capacity)
6312: 1994	Polyethylene containers for the transport of materials — Specification (*second revision*)
2771 (Part 1) : 1990	Corrugated fibreboard boxes — Specification: Part 1 General requirements (*second revision*)
10627 : 1983	Methods of sampling of pesticidal formulations
16674 : 2018	Spiromesifen, technical — Specification

3 REQUIREMENTS

3.1 Constituents

3.1.1 The material shall consist of spiromesifen technical, suspended in suitable solvent(s) and emulsifiers, stabilizers, antifoaming agents and preservatives.

3.1.2 Spiromesifen technical, employed in the formulation of this material, shall conform to IS 16674 : 2018.

3.2 Physical

The material shall comply with the following physical requirements:

3.2.1 *Description*

The material shall be in the form of light grey suspension liquid.

3.2.2 *Pourability*

When determined by the method prescribed in Annex B**,** the residue shall not be more than 5 percent (*m/m*) and, rinsed residue shall not be more than 0.25 percent (*m/m*).

3.2.3 *Spontaneity of Dispersion*

When determined by the method prescribed in Annex C**,** the spontaneity of dispersion (dispersibility) shall not be less than 90 percent (*m/m*).

3.2.4 *Suspensibility*

When determined by the method prescribed in Annex D**,** the suspensibility shall be minimum 90 percent (*m/m*).

3.2.5 *Wet Sieve Test*

Not less than 90 percent by mass of the material shall pass through 75 microns IS sieve [*See* IS 460 (Part 1)], when determined by the method prescribed in **1.1.1** of IS 6940.

3.2.6 *Persistent Foam*

When determined by the method prescribed in Annex E, the persistent foaming shall be 15 ml, maximum after 1 minute.

3.3 Chemical

The material shall comply with the following chemical requirements:

3.3.1 *Spiromesifen Content*

When determined by the method prescribed in Annex A, the observed spiromesifen content, percent by mass of any of the samples shall not differ from the declared nominal value by more than tolerance limits indicated below:

Nominal Value, Percent	*Tolerance limits, Percent of the Nominal Value*
Up to 9	+10
	−5
Above 9 and below 50	± 5
50 and above	+ 5
	-3

3.3.1.1 The actual value of spiromesifen content in the formulation shall be calculated to the second decimal place and then rounded off to first decimal place before applying the tolerances given in **3.3.1**.

3.3.2 *Acidity*

When determined by the method prescribed in **13.5.2** of IS 6940, the acidity [as sulphuric acid (H_2SO_4)] shall not be more than 0.5 percent (*m/m*).

4 TESTS

4.1 Tests shall be carried out as prescribed in **3.2** and **3.3**.

4.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5 PACKING

The material shall be packed in high density polyethylene (HDPE) containers conforming to IS 9754. These shall be further packed in corrugated fiberboard boxes conforming to IS 2771 (Part 1). All the containers shall also comply with the general requirements as stipulated in IS 8190 (Part 2).

6 MARKING

6.1 The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to any other information as required under the *Insecticides Act*, 1968 and Rules framed there under:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal spiromesifen content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules,* 2011.

6.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

6.2.1 The use of the standard mark is governed by the provisions of *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made thereunder. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

7.1 When freshly manufactured material in bulk quantity and/or the retail pack of the formulated product is offered for inspection, representative sample of the materials shall be drawn as prescribed in IS 10627, and if tested within 90 days of its date of manufacture, the criteria for conformity shall be the contents in percent (*m/m*), shall not be less than the declared nominal value. The upper limit for conformity shall be the same as those given in clause **3.3.1** of this standard.

7.2 When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627, however, the criteria for conformity of the material, when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under **3.3.1** of this standard.

ANNEX A

(*Clause* 3.3.1)

DETERMINATION OF SPIROMESIFEN CONTENT

A-1 PRINCIPLE

Spiromesifen content is determined by High Performance Liquid chromatography (HPLC) with UV-VIS detector using internal standard technique.

A-2 APPATATUS

A-2.1 High performance liquid chromatograph (HPLC) UV-VIS detector coupled to a printer-plotter-cum-integrator. The operative conditions suggested below are typical, which can be changed provided the standardization is done.

Colum	Stainless steel, C-18, 5µm, 25 cm, length, 4.0 mm i.d.
Detector	UV detector (260 nm)
Mobile phase	Acetonitrile : Water (80 : 20 v/v)
Flow rate	1.0 ml/minute.
Injection volume	20µl

A-2.2 Microliter Syringe — 5/20µl

A-2.3 Glasswares

A-3 REAGENTS

A-3.1 Acetonitrile HPLC grade equivalent

A-3.2 Water HPLC grade or equivalent

A-3.3 Internal Standard — Propyl benzoate AR or equivalent.

A-3.4 Spiromesifen reference standard of known purity.

A-4 PROCEDURE

A-4.1 Preparation of Mobile Phase

Mix acetonitrile and water in the proportion of 80 : 20 (v/v) and pass through a membrane filter under vacuum. Homogenize the mixture using a magnetic stirrer and allow to stand at the room temperature.

A-4.2 Preparation of Internal Standard Solution

Weigh out accurately 1.0 g of propyl benzoate into, a 100 ml volumetric flask and make up the volume up to the mark with acetonitrile. Shake well to homogenize.

A-4.3 Preparation of Standard Solution

Weigh out accurately 0.25 g of spiromesifen reference standard of known purity into a 100 ml volumetric flask. Dissolve the content with acetonitrile. (If required keep the flask in a ultrasonic bath for 10 min), and make up the volume up to the mark with acetonitrile (Solution A).

Pipette out 20 ml of Solution A into another 100 ml volumetric flask and add 10 ml of internal standard solution (Refer **A-4.2**) and make up the volume with the acetonitrile shake well to homogenize. This is standard solution (Solution B)

A-4.4 Preparation of Sample Solution

Weigh out accurately a quantity of a sample equivalent to 0.25 g of spiromesifen (approx.1.0 g) into a 100 ml volumetric flask and add 10 ml of water (HPLC grade or equivalent). Keep the flask for 10 min and make up the volume up to the mark with acetonitrile (Solution C). Pipette out 20 ml of the solution B into another 100 ml volumetric flask and add it to 10 ml, of internal standard solution (Refer **A-4.2**) and make up the volume with the acetonitrile. Shake well to homogenize. This is sample solution (Solution D)

A-4.5 Estimation

A-4.5.1 Inject 20µl of reference standard solution (Ref : **A-4.3**) into the HPLC followed by the sample solution (Ref : **A-4.4**).

From the chromatograms of standard solution and sample solution, measure the peak areas of spiromesifen and internal standard peaks and compute the percentage of spiromesifen as given in **A-5**.

[Note: Use guard column for reverse phase and, change the guard column when the peak shape gets disturbed.]

A-4.5.2 Retention times (Guide Values) :

Internal standard (Propyl benzoate)	- 3.34 minutes.
Spiromesifen	- 5.00 minutes.
Total run time	- 8.00 minutes.

A-5 CALCULATON

$$\textit{Spiromesifen content, percent by mass} = \frac{M_1 \times A_3 \times A_2 \times p}{M_2 \times A_4 \times A_1}$$

where,

M_1 = Mass in g of standard spiromesifen in standard solution;

M_2 = Mass in g of the sample taken for test;

A_1 = Peak area of spiromesifen in the chromatogram of standard solution;

A_2 = Peak area of spiromesifen, in the chromatogram of sample solution;

A_3 = Peak area of internal standard, in the chromatogram of standard solution;

A_4 = Peak area of internal standard, in the chromatogram of sample solution;

p = Percentage of purity, in the spiromesifen reference standard.

ANNEX B

(*Clause* 3.2.2)

DETERMINATION OF POURABILITY

B.1 APPARATUS

Five hundred ml stoppered cylinder conforming to ISO 4783. The requirement for this are volume equivalent to 1 subdivision of the scale of 5 ml, capacity corresponding to lowest graduation scale of 50 ml, capacity corresponding to highest graduation mark of 500 ml , length of scale of 250 mm, overall height of 39 cm, diameter of the base of 10 cm and, stopper of B 34.

NOTE — High density polyethylene bottles (1000 ml volume) and Kilner jars (700 ml volume) can be used out but, this must be recorded with result.

B.2 PROCEDURE

Weigh the empty cylinder along with stopper (W_0 g) and add enough of the sample to be tested in a cylinder to leave approximately 20 percent of the volume of the cylinder usage. Stopper the cylinder and weigh it (W_1 g). Allow the cylinder to stand undisturbed for 24 h and then pour out the sample for 60 s at an angle of 45° and then finally invert the cylinder for 60 s. Reweigh the cylinder along with stopper (W_2 g).

Add distilled water at 20º C (a volume of 80 percent of that cylinder). Stopper the cylinder invert the cylinder 10 times(Cylinder's vertical axis is turned through 180 degrees and, then brought back to original position). Then empty the cylinder as before. Weigh the cylinder along with stopper (W_3 g). Calculate the residue (R) and, rinsed residue (R") as follows:

$$\textit{Percent residue} = \frac{W_2 - W_0}{W_1 - W_0} \times 100$$

$$\textit{Percent rinsed residue} = \frac{W_3 - W_0}{W_1 - W_0} \times 100$$

ANNEX C

(*Clause* 3.2.3)

DETERMINATION OF SPONTANEITY OF DISPERSION (DISPERSIBILITY)

C-1 SPONTANEITY OF DISPERSION

When determined by the method prescribed below, the spontaneity of dispersion shall not be less than 80 percent by mass.

C-2 PROCEDURE

Take 900 ml of standard hard water (342 ppm), in a beaker and keep the beaker on magnetic stirrer. Add 8.8 g to 9.2 g of sample in the stirred water and continue the stirring for 1 min. Then switch of stirrer and allow the suspension to stand undisturbed for 1 min.

Withdraw, by means of a vacuum pump, nine-tenth (810 ml) of the suspension, determine by gravimetric means the solid obtained, in the remaining 90 ml in the beaker. This can be done most conveniently by evaporating the liquid in the rotary vacuum evaporator and drying to constant weigh.

C-3 CALCULATION

$$\textit{Dispersibility \% m/m} = \frac{(m-W)}{m} \times 111.1$$

where,

W = mass in g of residue after drying; and

m = mass in g of sample taken.

ANNEX D

(*Clause* 3.2.4)

DETERMINATION OF SUSPENSIBILITY

D-1 PRINCIPLE

Spiromesifen content is determined by High Performance Liquid chromatography (HPLC) with UV-VIS detector, using internal standard technique.

D-2 APPARATUS

D-2.1 High Performance Liquid chromatograph (HPLC) equipped with UV-VIS detector and, coupled to a printer-plotter-cum-integrator. The operative conditions suggested below are typical, which can be changed provided the standardization is done.

Column	Stainless steel, C-18,5 µm, 25 cm length, 4.0 mm i.d
Detector	UV detector (260nm)
Mobile phase	Acetonitrile : Water (80 : 20 v/v)
Flow rate	1.0 ml/min
Injection volume	20 µl

D-2.2 Microliter syringe — 5/20 µl

D-2.3 Glasswares

D-3 REAGENTS

D-3.1 Acetonitrile HPLC grade or equivalent.

D-3.2 Water HPLC grade or equivalent.

D-3.3 Internal Standard — Propyl benzoate AR grade or equivalent.

D-3.4 Spiromesifen reference standard of known purity.

D-4 PROCEDURE

D-4.1 Preparation of Mobile Phase

Mix acetonitrile and water in the proportion of 80 : 20 (v/v) and, pass through a membrane filter under vacuum. Homogenize the mixture using a magnetic stirrer and allow to attain the room temperature.

D-4.2 Preparation of Internal Standard Solution

Weigh out accurately 1.0 g of propyl benzoate into a 100 ml volumetric flask and make up the volume up to the mark with a acetonitrile. Shake well to homogenize.

D-4.3 Preparation of Standard Solution

Weigh out accurately 0.05 g of spiromesifen reference standard of known purity into a 100 ml volumetric flask. Dissolve the content with acetonitrile (If required keep the flask in an ultrasonic bath for 10 min). Add 10 ml of internal standard solution (Refer **A-4.2**) and make up the volume up to the mark with acetonitrile (Solution A).

D-4.4 Preparation of Sample Solution of Suspensibility

For carrying out suspensibility test, start with 0.25 g of the sample, add 30 ml standard hard water and proceed as described in 11.2 of IS 6940 : 1982. Transfer the bottom 25 ml of the suspension in 100 ml volumetric flask containing 10 ml of internal standard (Refer **A-4.2**). Make up the volume up to the mark with acetonitrile. This is sample solution (Solution B).

D-4.5 Estimation

Inject 20 µl of reference standard solution into the HPLC followed by the sample solution from the chromatograms of standard solution and sample solution measure the peak areas of sipromesifen and internal standard peaks and compute the spiromesifen, in the suspension.

D-5 CALCULATION

Calculate the spiromesifen content in the suspension, including the sediment by the following formula:

$$\textit{Spiromesifen content (in g) in the suspension including sediment} = \frac{A_3 \times A_2 \times m_1 \times p}{A_4 \times A_1 \times 100}$$

where,

A_1 = Area of the spiromesifen peak in the standard solution;

A_2 = Area of the spiromesifen peak in the sample solution;

A_3 = Area of the internal standard peak in the standard solution;

A_4 = Area of the internal standard peak in the sample solution;

m_1 = Mass in g of spiromesifen standard reference solution; and

p = Percent of purity, in sipromesifen reference standard.

$$\textit{Suspensibility}\ \%\ \frac{m}{m} = \frac{1\,000\,(M-m)}{9M} = \frac{(M-m)}{M} \times 111.1$$

where,

M = Mass in g of spiromesifen present in the sample taken for suspensibility test; and

m = Mass in g of spiromesifen present in the suspension including sediment.

$$M = \frac{m_2 \times \textit{Observed Spiromesifen A.I.of SC}}{100}$$

Where,

m_2 = Mass in g of spriomesifen SC taken for test.

ANNEX E

(*Clause* 3.2.6)

DETERMINATION OF PERSISTENT FOAMING

E-0 SCOPE

The method is suitable for determining the foam created, when suspension concentrates are diluted.

E-1 OUTLINE OF METHOD

The suspension concentrate is diluted in a measuring cylinder of standard dimensions which is inverted 30 times and, the amount of foam created and remaining after certain times is measured.

E-2 REAGENT

E-2.1 Standard hard water (500 ppm)

E-3 APPARATUS

E-3.1 Graduated Cylinder

Glass stoppered of 250 ml capacity, with 2 ml graduations. The distance between the 0 mark and the 250 ml mark should be 20 to 21.5 cm and between the 250 ml mark and the bottom of the stopper, 6-8 cm (*see* Note 1).

E-3.2 Stopwatch

E-4 PROCEDURE

The mass of sample to be taken is that mass required to make 200 ml of a suspension with a concentration recommended in the directions for use supplied with the product. Where, several concentrations are recommended, the maximum concentration shall be used.

Put about 180 ml of standard hard water into the 250 ml measuring cylinder kept on a top pan balance and weigh in the required amount of suspension concentrate. Top up with standard hard water up to 200 ml mark. Stopper the cylinder and invert 30 times (*see* Note 2). Place the stoppered cylinder upright on the bench and immediately start the stopwatch. Read the volume of foam produced and remaining after 1 minute (*see* Note 3).

NOTES

1 The cylinder should be clean and free from grease.

2 The expression ‘invert the cylinder’ means that the stoppered cylinder is held by 2 hands, one at each end of the cylinder, which are insulated from the cylinder by means of a cloth. The upright cylinder is turned through 180°C and back to its original position without any ‘bounce’ occurring, this operation taking approximately 2 seconds. It is convenient to observe a stop-clock equipped with a second hand which sweeps once every 60 seconds, while doing this.

3 A few bubbles round the periphery are not significant. Any volumes above the 250 ml mark should be marked on the outside and the volume of foam thus determined.

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Amendments Issued Since Publication

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